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(56) Documents cited

GB 2211196 A	GB 1307637 A	EP 0347220 A1
US 4765916 A	US 4062451 A	

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(54) Soluble sachets

(57) The invention provides a package which comprises a hazardous chemical dissolved or dispersed in a liquid or gel contained in a water-soluble or water-dispersible laminated film bag, sachet or container. The chemical comprises an agrochemical such as a pesticide, herbicide.

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SOLUBLE SACHETS

This invention relates to a package comprising a liquid chemical or chemical dissolved or dispersed in a liquid or gel which is contained in an envelope of water-
5 soluble or water-dispersible material.

At present, most hazardous liquids are stored in metal drums or, where smaller quantities are required, plastic containers.

Hazardous compounds, especially agrochemical compounds,
10 are formulated in a variety of ways. It is however especially desirable for farmers to handle such compounds when they are in liquid form. This facilitates the spreading of such compounds. There are however difficulties and drawbacks in handling liquids. The
15 liquids can be spilt on the ground or may leak due to holes in the containers. The containers may also rupture when subjected to a physical shock.

It is thus difficult to devise a containing system which is suitable for farmers, and safe for all those
20 handling the containers, and also safe for the environment.

It is known that agrochemicals may be contained in soluble bags or sachets made from films. However, such films may crack and break and thus cause spillage of the agrochemicals they contain. In fact, there are a variety
25 of defects which may be present in films, which lead to weaknesses of the film and consequently a potential source

of leakage. The presence of air bubbles, of dust particles or other foreign bodies, of gel particles or of thin points on or in the film are all potential weak points. If a film with such a weak point is subjected to a lot of handling or physical shock, the film may fail at that point. This is especially a problem in the agrochemical industry where containers may be subjected to rough or unsafe handling by distributors or farmers.

The weaknesses of films mentioned above are present to a greater or lesser extent in certain types of films, depending upon their method of manufacture. When films are made by casting, there may be fewer pinholes, but there are more often tiny pieces of gel inclusions. When films are made by extrusion, there are more pinholes.

The present invention seeks to provide a new container system for agrochemicals which is safe to handle.

The invention further seeks to provide a new container system for agrochemicals which is convenient for endusers, eg farmers, to handle.

The invention further seeks to provide a new container system for agrochemicals which reduces the risks of pollution and environmental damage.

In one aspect, the invention seeks to avoid leakage of liquid or dissolved chemicals through the pinholes of a film container system. Although pinholes are generally rare, even the presence of one pinhole in many thousands of

containers is enough to cause damage, since the liquid in the container passes through the pinhole and contaminates the surrounding environment.

Other objects and advantages of the invention will
5 be apparent from the description which follows.

The container system of the invention is characterised in that a hazardous material in a liquid or gel form is contained in a water-soluble or water-dispersible bag made of a laminated film.

10 According to a preferred feature of the invention the hazardous compounds are agrochemicals, eg pesticides or plant protection agents or plant growth regulators.

The present invention accordingly provides a package which comprises a hazardous chemical dissolved or
15 dispersed in a liquid or gel contained in a water-soluble or water-dispersible laminated film.

In this specification the term laminated film means a film which has been made from two or more originally separated layers which are joined together. The layers may
20 be joined together in the laminate by known methods. For example, the layers or the laminate may be joined by pressure, heating, crosslinking, fusion, adhesion or any combination thereof. Adhesion of the layers may be obtained through the use of a separate adhesive, or, when
25 appropriate, water.

The laminated film is preferably completely, or

substantially completely, dispersible or, more preferably, soluble in water.

When suitable water-soluble or water-dispersible layers are used, a particularly convenient way to obtain a
5 laminated film is by adhesion of the separate layers, either using adhesive PVA (generally of low molecular weight) and/or more simply by means of water.

Because two or more layers are used to produce the laminated film the chance of pinholes occurring in the film
10 is reduced to almost nil. This is because it is unlikely that two pinholes in separate layers will overlap.

Furthermore, the tensile strength of a multi (eg two) layer film is better than the tensile strength of a similar film or the same thickness which is made of a single layer.

15 The laminated films which are used in the invention generally have a thickness of from 10 to 250 microns, preferably from 15 to 80 microns. The individual layers constituting the laminated films which are used in the invention are generally each half of these figures. When
20 two layers are used, the ratio of thickness of the two layers is generally from 0.1 to 10, preferably from 0.5 to 2. Bags or sachets made of laminated films according to the invention generally have a content of from 0.2 to 12 litres, preferably from 0.45 to 6 litres.

25 The materials which may be used in the invention are water-soluble or water-dispersible materials which are

insoluble in organic solvents used to dissolve or disperse the chemicals they are used to contain. Suitable materials include polyethylene oxide or methyl cellulose, but preferably the material comprises or is derived from
5 polyvinyl alcohol, ie partially or fully alcoholysed or hydrolysed for example 40-99%, preferably 70-92% alcoholysed or hydrolysed polyvinyl acetate films.

The layers of the laminated films of the invention may be made of the same material or of different materials.
10 Films made from layers of different materials may have advantageous properties. For example, an inner layer of a package may be made more resistant to, eg, the agrochemical it contains. In addition, the outer layer of the bag can be selected to have one or more of the following
15 properties:

- (i) to dissolve more quickly (compared to an inner layer or a single layer package) in water,
- (ii) to have improved mechanical properties including improved resistance to mechanical
20 damage,
- (iii) to have improved machinability,
- (iv) to have less susceptibility to relative humidity,
- (v) to have resistance to freezing and/or high
25 temperatures.

One or more of the layers of the laminated film may

contain a plasticiser. A suitable plasticiser content in the inner layer may improve the sealing properties of the film, and make the film less likely to stretch. The film will thus be easier to process on machinery and to seal
5 around the hazardous liquid. A suitable plasticiser content in the outside layer of the container makes the outer surface more flexible and thus more resistant to physical damage from low temperature or shock and movement.

The layers of the laminated film may be made using
10 different techniques, eg extrusion or casting. A laminate made from layers produced by different methods may have advantageous properties including greater flexibility, increased strength and increased resistance to stretching. The increased strength and/or resistance to stretching may
15 be in one direction in the film. The laminated films for use in the present invention are used to produce packages which avoid the time-consuming and hazardous consequences of prior art packages which leak in the filling process or during subsequent handling due to film defects.

20 The packages according to the invention, as for example bags or containers may contain either liquids or gels. The packages preferably contain a gel.

According to a particular feature of the invention, the gels are chosen in such a way that one or more of the
25 following features are present:

- * the resulting gels form a continuous system; and/or

- * the resulting gels have a viscosity of 500 to 50,000 centipoises, more preferably of 1000 to 12,000 centipoises (these viscosities are Brookfield viscosities measured at room temperature with a viscosimeter in the form of a flat plate rotating at 20 revolutions per minute); and/or
- * the gel has a phase difference ϕ between the controlled shear stress and the resulting shear strain such that $\tan(\phi)$ is less than or equal to 1.5, preferably less than or equal to 1.2. $\tan(\phi)$ is the tangent of the angle ϕ (or phase difference). The measurement of ϕ is made at room temperature by means of a rheometer having a flat fixed plate with a rotating cone above this plate such that the angle between them is less than 10° , preferably 4° . The cone is caused to rotate by means of a controlled speed motor; the rotation is a sinusoidal one, ie the torque and the angular displacement change as a sine function with time. This angular displacement corresponds to the above-mentioned shear strain; the torque of the controlled speed motor (which causes the angular displacement) corresponds to the above-mentioned controlled shear stress; and/or
- * the gels preferably have a density greater than 1, preferably greater than 1.1; and/or

- * the gels have a spontaneity (as hereinafter defined) less than 75, preferably less than 25.

The spontaneity is assessed according to the following method: a mixture of 1 ml gel with 99 ml water
5 are put into a 150 ml glass tube (diameter 22 mm) which is stoppered and inverted through 180° (upside down) at room temperature. The number of times required to completely disperse the gel is called the spontaneity.

By the term "continuous system", is meant a
10 material which is visually homogeneous, that is to say which has the visual appearance of having only one physical phase; this does not exclude the possibility of small solid particles being dispersed therein, provided these particles are small enough not to constitute a visible separate
15 physical phase.

It is known that a gel is generally a colloid in which the dispersed phase has combined with the continuous phase to produce a viscous, jelly-like product; it may also be a dispersed system typically comprising a high molecular
20 weight compound or aggregate of small particles in very close association with a liquid.

In order to make a bag, the film needs to be shaped (and possibly partially sealed) and then filled with the gel. Generally the gels are able to flow, although
25 possibly at a slow rate due to their high viscosity. A container which is used to contain the gels cannot be

easily emptied due to this high gel viscosity (a reason why the gels were not used up to now in the agriculture). When filled, the bags have to be finally sealed, generally heat sealed, to be closed.

5 The following Examples illustrate the invention. In the Examples, unless otherwise specified, percentages are by weight.

EXAMPLE 1

A film in a roll form is constructed from two
10 thinner films by lamination: both films are made from an 88% hydrolysed polyvinyl alcohol (cold water soluble), each 25 microns in thickness; one has a 17% plasticiser content, the other has 15% plasticiser content. The two films are laminated together with heat (100°C) and pressure to form
15 one film of 50 microns thickness.

This film is then used to produce 1 litre sachets containing a solvent based liquid herbicide (a mixture of ioxynil and bromoxynil esters) by using "form-and-fill" methods. The herbicide is a solution in a C10 aromatic
20 hydrocarbons mixture as solvent.

The film is placed on the machine so that the high plasticiser layer is produced on the outside of the sachets. The film is thus easy to process.

No leak is observed during the manufacture,
25 handling, and transporting of 500 sachets.

EXAMPLE 2

A gel is made by stirring at 50°C a mixture of:

Active ingredient: 2,4-D (a phenoxy benzoic acid
isooctyl ester): 64.8%

5 Solvent: aromatic solvent with flash point of
65°C: 24.2%

Surfactant: a mixture of
a non ionic/sulfonate blended
emulsifier 4%

10 and calcium alkylbenzene sulfonate 1%

Gelling agent: a mixture of dioctylsulfosuccinate salt
and sodium benzoate: 6%

The mixture is stirred and shaken, with heating,
until each component is dissolved or dispersed.

15 During stirring, dissolution takes place, and
thereafter gelation. Gelation is increased during cooling
to room temperature (20°C).

The Brookfield viscosity of the gel is 3000
centipoises. The emulsion stability is good in the above
20 described test.

1100 g of this gel are put in a 1 litre bag made of
a film of PVA similar to the film of Example 1. The bag,
which is almost full (about 95% v/v), is heat sealed. The
density both of the gel and of the bag containing the gel
25 is 1.1.

The bag is then dropped 10 times from 1.2m upon the

ground. No breaking or leakage is observed.

Another bag made in the same way as the previous one is tested for pinhole protection. A needle (diameter 0.6mm) is passed through the bag. A small droplet is
5 observed which forms at the locus where the needle passed, but this droplet was small enough not to drop from the bag and not to flow along the bag.

CLAIMS

1. A package which comprises a hazardous chemical dissolved or dispersed in a liquid or gel contained in a water-soluble or water-dispersible laminated film.

5 2. A package according to claim 1 wherein the hazardous chemical is an agrochemical or a pesticide or a plant protection agent or a plant growth regulator.

 3. A package according to claim 1 or 2 wherein the hazardous chemical is dissolved or dispersed in a
10 liquid.

 4. A package according to claim 1 or 2 wherein the hazardous chemical is dissolved or dispersed in a gel.

 5. A package according to any one of the preceding claims wherein the laminated film has a thickness
15 of from 10 to 250 microns.

 6. A package according to any one of the preceding claims wherein the laminated film has a thickness of from 15 to 80 microns.

 7. A package according to any one of the
20 preceding claims which has a content of hazardous chemical of from 0.2 to 12 litres.

 8. A package according to any one of the preceding claims which has a content of hazardous chemical of from 0.45 to 6 litres.

9. A package according to any one of the preceding claims wherein the water-soluble or water-dispersible laminated film is insoluble in the liquid or gel used to dissolve or disperse the hazardous chemical.

5 10. A package according to any one of the preceding claims wherein at least one of the layers of the film is selected from polyethylene oxide, methyl cellulose or partially or fully alcoholysed or hydrolysed polyvinyl acetate.

10 11. A package according to claim 10 wherein the said at least one layer is 40-99% alcoholysed or hydrolysed polyvinyl acetate.

 12. A package according to claim 10 wherein the said at least one layer is 70-92% alcoholysed or hydrolysed
15 polyvinyl acetate.

 13. A package according to any one of the preceding claims wherein at least two of the layers of the laminated film are made of the same material.

 14. A package according to any one of claims 1 to
20 11 wherein at least two of the layers of the laminated film are made of a different material.

 15. A package according to any one of the preceding claims wherein the laminated film consists essentially of two layers.

25 16. A package according to claim 15 wherein the ratio of the thickness of the two layers is from 0.1 to 10.

17. A package according to claim 15 wherein the ratio of the thickness of the two layers is from 0.5 to 2.

18. A package according to any one of claims 4 to 17 wherein the gel has a viscosity of 500 to 50,000
5 centipoises.

19. A package according to any one of claims 4 to 17 wherein the gel has a viscosity of 1000 to 12,000 centipoises.

20. A package according to any one of claims 4 to
10 18 wherein the gel has a phase difference ϕ between the controlled shear stress and the resulting shear strain such that $\tan(\phi)$ is less than or equal to 1.5.

21. A package according to any one of claims 4 to
15 18 wherein the gel has a phase difference ϕ between the controlled shear stress and the resulting shear strain such that $\tan(\phi)$ is less than or equal to 1.2.

22. A package according to any one of claims 4 to 21 wherein the gel has a spontaneity less than 75.

23. A package according to any one of claims 4 to
20 21 wherein the gel has a spontaneity less than 25.

24. A process for the production of a package according to any one of the preceding claims which comprises laminating two or more layers together by pressure, heating, crosslinking, fusion or by means of
25 water to obtain the laminated film.

25. A package according to claim 1 substantially

as hereinbefore described.

26. A process according to claim 24 substantially
as hereinbefore described.

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